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FY93 End of Fiscal Year Letter
(01 Oct 1992 - 30 Sep 1993)

ONR CONTRACT INFORMATION

Contract Title: Microstructure and Texture Control of Fatigue in Hydrogen and
Marine Environments
Performing Organization: Chemical Engineering and Materials Science, Univ. of
Minnesota, Minneapolis, MN 55455
Principal Investigator: William W. Gerberich
Contract Number: N00014-89-J-1726
R & T Project Number: 4311934
ONR Scientific Officer: Dr. George Yoder

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Enclosure (1)

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A. Scientific Research Goals

It is currently not understood how the anisotropic elastic, plastic and fracture characteristics interact with environment to limit fatigue life in a corrosive medium. This program examines how texture and anisotropic slip character interact with environment in two general classes of BCC iron base and HCP titanium base alloy systems. Selected area electron channeling, transmission electron microscopy, atomic force microscopy, and fracture micromechanic methods are combined to evaluate microstructural effects on environmentally-induced fatigue initiation and growth. In addition, as it is essential to understand the transition from small crack to long crack behavior, additional threshold studies on both Fe-Si and HSLA polycrystals need to be accomplished. In this way, the various stages of nucleation and growth may be combined together for life prediction. The objective is to provide a partial basis for alloy and materials processing design against fatigue in a marine environment.

B. Significant Results

We are now taking a step-wise approach in attempting to understand the corrosion fatigue life of high strength low alloy steels and Ti-6Al-4V. Considerable findings have been made in the both alloy systems. In the previous year, we had discussed ways of quantifying the various stages of fatigue life, as given by

$$N_{total} = N_i + N_{small} + N_{long}$$

where N_i is the number of cycles to initiation, N_{small} is the number spent growing the crack in the small crack regime to where it will grow as a long crack and N_{long} is the normal number of cycles integrated from the long crack threshold to the critical crack size. Of most importance to this project, is N_i , where it was proposed that N_i could also be interpreted in terms of an effective threshold stress intensity, ΔK_{th}^{eff} . Since ΔK_{th}^{eff} can be related to a modified Griffith concept through

$$K_I^{cr} = \frac{1}{\beta'} \exp \left\{ \frac{k_{IG}^2}{\alpha'' \sigma_{ys}} \right\} \quad (1)$$

in principal, one can use direct computational means to define β' and α'' for a given material system and in turn use equation (1) to define the effective threshold stress intensity in fatigue. One of the significant results this year is in the partial proof of Eq. (1) as applied to fatigue. We had previously shown that Eq. (1) was applicable to the ductile-brittle transition in Fe-3wt%Si single crystals, with $\beta' = 20 \text{ MPa}^{-1} \text{m}^{-1/2}$ and $\alpha'' = 3.1 \times 10^{-4} \text{ MPa}\cdot\text{m}$. With $k_{IG} = 0.85 \text{ MPa}\cdot\text{m}^{1/2}$ and $\sigma_{ys} = 300 \text{ MPa}$ this gave a critical K_{Ic} of $118 \text{ MPa}\cdot\text{m}^{1/2}$ only slightly higher than the value of $\sim 90 \text{ MPa}\cdot\text{m}^{1/2}$ observed at room temperature. By dropping k_{IG} to $k_{IG}^H = 0.75 \text{ MPa}\cdot\text{m}^{1/2}$ due to hydrogen, a value of $K_{I_{th}} = 21 \text{ MPa}\cdot\text{m}^{1/2}$, is only slightly larger than the sustained load threshold of $17 \text{ MPa}\cdot\text{m}^{1/2}$ observed. This was extensively discussed at the Parkins Symposium.¹ The importance to this study is that if σ_{ys} is increased during fatigue loading near threshold, *the threshold value continuously decreases as the local flow stress increases from cyclic hardening*. By allowing the cyclic flow stress to increase from 300 to 400 MPa, the threshold stress intensity should decrease to $\Delta K_{th}^H = 4.7 \text{ MPa}\cdot\text{m}^{1/2}$ according to Eq. (1). We have confirmed this with the following critical experiment. We measured the sustained load threshold in hydrogen to be $17 \text{ MPa}\cdot\text{m}^{1/2}$. A companion specimen was then taken and cycled at $8 \text{ MPa}\cdot\text{m}^{1/2}$ in flowing nitrogen at 253 K to avoid any environmental interactions. When this was subsequently held at

a sustained load of $8 \text{ MPa}\cdot\text{m}^{1/2}$ in hydrogen, the crack grew. Subsequent tests with the cycling and sustained stress intensities at 5 and $4 \text{ MPa}\cdot\text{m}^{1/2}$ showed growth at the former but not in the latter. Thus, the sustained load threshold stress intensity has been dropped from 17 to about $4\text{--}5 \text{ MPa}\cdot\text{m}^{1/2}$ by prior fatigue work hardening of the local region about the crack tip. These results were presented in more detail at the Fatigue '93 meeting in Montreal.

The second significant result is that we have been able to extend the atomic force microscopy results on fatigue surface slip upsets as an initiation criterion. We had shown previously that one could describe the number of cycles to initiation by the surface slip spacing, h_s , the threshold stress intensity, ΔK_{th} , and the fraction, f , of reverse plastic strain, $\Delta\epsilon_p$, going into producing slip upset at the surface. This gave

$$N_i = \frac{\Delta K_{th}^2}{4\sigma_{ys} E f \Delta\epsilon_p h_s} \quad (2)$$

and is currently a part of a paper submitted to *Acta Metall. et Mater.*² We have more recent results which show that the fraction of plasticity going into producing surface damage decreases with increasing numbers of cycles. This in fact should be expected since greater dislocation densities at larger cumulative plastic strains should produce both more annihilation and more low energy dislocation structures. Thus, the rate of damage accumulation should decrease. The bottom line is that since f is also a function of N , e.g. $\propto 1/N^{1/2}$, then it can be shown that Eq. (2) reduces to the Manson Coffin equation. We are still collecting data on this but the implications are enormous if true. For one, it would give a crystallographic and material rationale as to why the Manson-Coffin exponent varies between materials. It might also give a relatively inexpensive method for determining the Manson-Coffin exponent.

1. W.W. Gerberich, P. Marsh and H. Huang, in *Parkins Symposium on Fundamental Aspects of Stress Corrosion Cracking*, S. Bruemmer, et al., eds., TMS-AIME, Warrendale, PA (1992) pp. 191-204.
2. S.E. Harvey, P.G. Marsh and W.W. Gerberich, "Atomic Force Microscopy and Modeling of Fatigue Crack Initiation in Metals," submitted to *Acta Metall. et Mater.* (1993).

C. Future Work

Expectations are that such approaches will lead to a physically-based concept for total fatigue life. In this context, cyclic flow parameters, slip planarity, grain size and environmental susceptibility to crack initiation all become factors. Already we have successfully developed an experimental technique for estimating fatigue initiation life. We have also established that atomic force microscopy (AFM) of smooth bar surfaces is possible and should clarify and verify slip-step aspects of the initiation models. In addition, we have verified with a critical experiment that the threshold stress intensity approach, as affected by the cyclic hardening process, is a valid one for understanding hydrogen effects on corrosion fatigue thresholds. What needs to be accomplished next is the connectivity of this to fatigue initiation in aqueous solutions. Thus, future work will involve cyclic initiation life measurements, associated electron channeling of plastic strain and surface topography with AFM on materials subjected to aqueous environments.

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D. List of Publications/Reports/Presentations

1. Papers Published in Refereed Journals

Y. Katz, R.R. Keller, H. Huang and W.W. Gerberich, "A Dislocation Shielding Model for the Fracture of Semi-Brittle Polycrystals," *Metall. Trans. A* **29A** (1993) pp. 343-350.

W. Zielinski and R.R. Keller and W.W. Gerberich, "Heterogeneous Dislocation Loop Nucleation and Free Surface Effects on Plastic Deformation: An *in situ* TEM Study," *Mat'l's. Sci. and Engng. A* **164** (1993) pp. 196-200.

S. Harvey, H. Huang, S. Venkataraman and W.W. Gerberich, "Microscopy and Microindentation Mechanics of Single Crystal Fe-3wt%Si, Part I: Atomic Force Microscopy of a Small Indentation," *J. Materials Research* **8** No. 6 (1993) pp. 1291-1299.

T. Foecke, P.G. Marsh, N.R. Moody and W.W. Gerberich, "A Sample Fabrication-Induced Hydrogen Artifact in Fracture Experiments," *Scripta Metall. et Mater.* (1992).

P.G. Marsh and W.W. Gerberich, "A Microscopically-Shielded Griffith Criterion for Cleavage in Grain Oriented Silicon Steel," in press, *Acta Metall. Mater.* (1993).

2. Non-Refereed Publications and Published Technical Reports

(none)

3. Presentations

a. Invited

Invited Overview Presentation, "Hydrogen/Plasticity Interactions in Corrosion and Stress Corrosion," International Conference on Corrosion-Deformation Interactions, CD1 92, Fontainebleau, France, October 5-7, 1992.

Invited speaker, with S. Harvey, J. Hoehn and P.G. Marsh, "Atomic Force Microscopy of Hydrogen-Affected Slip Processes under Fatigue Conditions," in Fatigue of Advanced Materials Symposium, Spring TMS, Denver, February 24, 1993.

Invited speaker, with J.W. Hoehn, P.G. Marsh, S. Harvey and S. Venkataraman, "Fatigue Damage and Crack Initiation under Aggressive Environments," in Symposium in Crack Measurement and Life Prediction in Aggressive Environments, TMS, Pittsburgh, October 18, 1993.

b. Contributed

With P.G. Marsh, "Influence of Grain Size and Texture on Fatigue Crack Initiation of HSLA Steel in Hydrogen," TMS Fall Meeting, Chicago, November 4, 1992.

With N.R. Moody, J.E. Angelo and M.I. Baskes, "An Atomistic Study of Dislocation Emission Effects on Hydrogen Induced Fracture," Spring MRS Symposium W, San Francisco, April 12, 1993.

With P.G. Marsh, "Prediction of Hydrogen-Induced Fatigue Thresholds, ΔK_{th}^H , in Iron-Base Materials," Fatigue '93 Symposium, Montreal, Canada, May 6, 1993.

4. Books (and sections thereof)

W.W. Gerberich, P. Marsh, J. Hoehn, S. Venkataraman and H. Huang, "Hydrogen/Plasticity Interactions in Stress Corrosion Cracking," in *Corrosion-Deformation Interactions*, CD1 '92, T. Magnin, ed., *Les Editions de Physique Les Ulis*, France (1993) pp. 325-343.

P.G. Marsh and W.W. Gerberich, "Influence of Microstructure and Texture on Fatigue Crack Initiation in HSLA Steels in Hydrogen and Nitrogen Atmospheres," in *First Intern. Symp. on Microstructures and Mechanical Properties of Aging Materials*, P.K. Liaw, K.L. Murty, E.P. Simonen, R. Viswanathan and W.B. Jones, eds., TMS, Warrendale, PA (1993).

P.G. Marsh and W.W. Gerberich, "Prediction of Hydrogen-Induced Fatigue Thresholds, ΔK_{th}^H in Iron-Base Materials," in *Fatigue '93*, Vol. III, I. Dickson, J.P. Bailon, eds. (1993).

W.W. Gerberich, S.K. Venkataraman, J.W. Hoehn and P.G. Marsh, "Fracture Toughness of Intermetallics using a Micro-Mechanical Probe," in *Intern. Symp. on Structural Intermetallics*, TMS, Warrendale, PA (1993).

E. List of Honors/Awards

William W. Gerberich	Univ. of Minnesota	Plenary Lecturer (1 of 4) for Corrosion/Deformation Interaction '92, sponsored by Electricite de France, CNRS FRAMATOME; selected to be on '96 Organizing Committee.
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William W. Gerberich	Univ. of Minnesota	Elected Vice-Chairman of the Board of Governors for the NSF Sponsored Institute for Mechanics and Materials. The purpose of the board is to be an oversight body for the Institution's educational mission of fostering interdisciplinary understanding between the mechanics and materials communities.
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F. Participants (1992-93)

1. Dr. Yosef Katz, former Visiting Professor at the University of Minnesota and now at the Nuclear Research Establishment, Beer Sheva, Israel, is cooperating on a fatigue crack propagation study dealing with frequency and environmental effects..
2. Mr. Peter Marsh, responsible for the largest part of the electron channeling and fatigue interaction portions of the project is in his fourth year as a PhD candidate. He presented a major paper at Fatigue '93 in Montreal.
3. Ms. Stefanie Harvey joined the project last year to develop atomic force microscopy (AFM) techniques as a measure of fatigue initiation. She has passed her pre-lims and her work will be presented at an invited talk at the fatigue symposium during the Fall '93 TMS meeting in Pittsburgh.
4. Mr. Ron Birkhahn, a 2nd year M.S. student, will be finishing his degree, which deals principally with crack-tip strain measurements in HSLA steel by electron channeling.

G. Other Sponsored Research (1992-93)

1. DOE, Basic Energy Sciences Grant DE-FG02-84ER45141, "Micromechanisms of Brittle Fracture: STM, TEM and Electron Channeling Analysis," \$84,584, 7/1/92-6/30/93. 1/2 month summer salary.
2. DOE, Basic Energy Sciences Grant DE-FG02-88ER45337, "A Study of Scale Cracking and its Effect on Oxidation and Hot Corrosion," \$36,540, 11/1/92-10/31/93, 1/2 month summer salary.
3. NSF, Center for Interfacial Engineering (with 19 other P.I.'s), \$2,600,000, 10/1/92-9/30/93. support for 1 1/2 graduate students, 1/2 post-doctoral associate, no summer salary.
4. 3M/DARPA/ONR Grant N/N00014-92-J-1062, "Micromechanics of Interfaces in Metal Matrix Composites," \$50,000, 9/1/92-8/30/93, with Professor D. Kohlstedt, no summer salary.
5. Cardiac Pacemakers Inc., "Corrosion Studies of Biomedical Materials," \$42,000, 1/30/93-2/1/94, 1 month summer salary.
6. NSF, Center for Plasma Aided Manufacturing, 10/1/92-9/30/93, support for 2 co-advised students (Professors Pfender and Heberlein, Mechanical Engineering) no summer salary.

H. SUMMARY OF FY93
PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/PARTICIPANTS
(Number Only)

	<u>ONR</u>	<u>non ONR</u>
a. Number of Papers Submitted to Referred Journal but not yet published:	<u>2</u>	<u>15</u>
b. Number of Papers Published in Refereed Journals:	<u>4</u>	<u>10</u>
c. Number of Books or Chapters Submitted but not yet Published:	<u>4</u>	<u>8</u>
d. Number of Books or Chapters Published:	<u>2</u>	<u>5</u>
e. Number of Printed Technical Reports & Non-Referred Papers:	<u>-</u>	<u>-</u>
f. Number of Patents Filed:	<u>-</u>	<u>-</u>
g. Number of Patents Granted:	<u>-</u>	<u>1</u>
h. Number of Invited Presentations at Workshops or Prof. Society Meetings:	<u>3</u>	<u>2</u>
i. Number of Contributed Presentations at Workshops or Prof. Society Meetings:	<u>3</u>	<u>14</u>
j. Honors/Awards/Prizes for Contract/Grant Employees: (selected list attached)	<u>1</u>	<u>1</u>
k. Number of Graduate Students and Post-Docs Supported at least 25% this year on contract grant:	<u>3</u>	<u>9</u>
Grad Students: TOTAL	<u>3</u>	<u>9</u>
Female	<u>1</u>	<u>1</u>
Minority	<u>0</u>	<u>0</u>
Post Doc: TOTAL	<u>0</u>	<u>1</u>
Female	<u>0</u>	<u>0</u>
Minority	<u>0</u>	<u>0</u>
l. Number of Female or Minority PIs or CO-PIs		
New Female	<u>0</u>	<u>0</u>
Continuing Female	<u>0</u>	<u>0</u>
New Minority	<u>0</u>	<u>0</u>
Continuing Minority	<u>0</u>	<u>0</u>

Enclosure (4)